PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : **D21B 1/02, 1/04, D01C 1/00**	A1	(11) International Publication Number: **WO 99/41448** (43) International Publication Date: 19 August 1999 (19.08.99)

(21) International Application Number: PCT/US99/02643

(22) International Filing Date: 9 February 1999 (09.02.99)

(30) Priority Data:
09/021,888 11 February 1998 (11.02.98) US

(71) Applicant: NORTH CAROLINA STATE UNIVERSITY [US/US]; 1 Holladay Hall, Pullen Road, P.O. Box 7003, Raleigh, NC 27695-7003 (US).

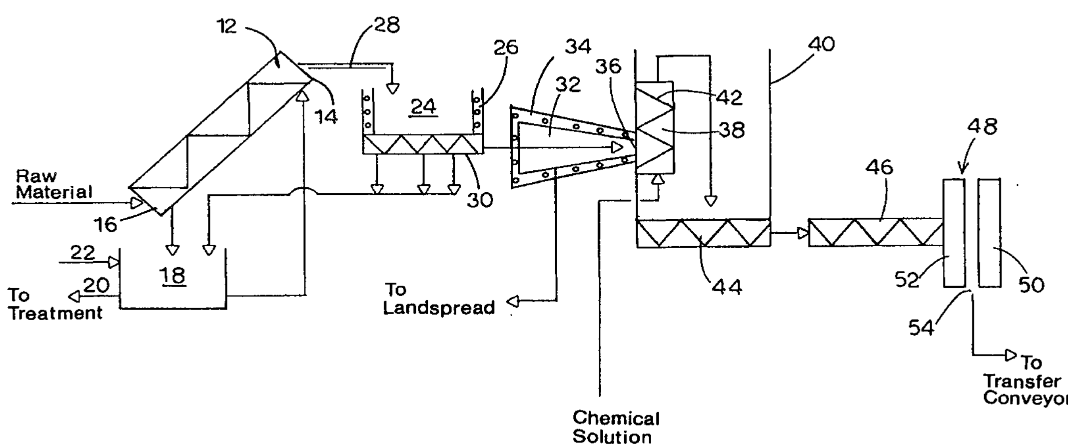
(72) Inventor: BYRD, Medwick, V., Jr.; 1321 Wake Forest Road, Raleigh, NC 27604 (US).

(74) Agents: JENKINS, Richard, E. et al.; Jenkins & Wilson, P.A., Suite 1400, University Tower, 3100 Tower Boulevard, Durham, NC 27707 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published
With international search report.
With amended claims.

(54) Title: METHOD FOR THE SIMPLE AND ENVIRONMENTALLY BENIGN PULPING OF NONWOOD FIBROUS MATERIALS



(57) Abstract

A process for producing a pulp suitable for papermaking from a nonwood fiber source material is described. Exemplary nonwood fiber source materials include kenaf and industrial hemp. The process includes the steps of washing the nonwood fiber source material with a washing solution in a washing screw (12); compressing the nonwood fiber source material to form a compressed plug in an impregnation screw (32); impregnating the compressed plug of the nonwood fiber source material with a pulping solution at ambient temperature and at atmospheric pressure in an impregnation vessel (38); feeding the impregnated nonwood fiber source material to form a papermaking pulp at a bottom port (54). Additional treatment of the papermaking pulp by acid chelation; by bleaching or by acid chelation and bleaching are also described.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania
AM	Armenia
AT	Austria
AU	Australia
AZ	Azerbaijan
BA	Bosnia and Herzegovina
BB	Barbados
BE	Belgium
BF	Burkina Faso
BG	Bulgaria
BJ	Benin
BR	Brazil
BY	Belarus
CA	Canada
CF	Central African Republic
CG	Congo
CH	Switzerland
CI	Côte d'Ivoire
CM	Cameroon
CN	China
CU	Cuba
CZ	Czech Republic
DE	Germany
DK	Denmark
EE	Estonia
ES	Spain
FI	Finland
FR	France
GA	Gabon
GB	United Kingdom
GE	Georgia
GH	Ghana
GN	Guinea
GR	Greece
HU	Hungary
IE	Ireland
IL	Israel
IS	Iceland
IT	Italy
JP	Japan
KE	Kenya
KG	Kyrgyzstan
KP	Democratic People's Republic of Korea
KR	Republic of Korea
KZ	Kazakstan
LC	Saint Lucia
LI	Liechtenstein
LK	Sri Lanka
LR	Liberia
LS	Lesotho
LT	Lithuania
LU	Luxembourg
LV	Latvia
MC	Monaco
MD	Republic of Moldova
MG	Madagascar
MK	The former Yugoslav Republic of Macedonia
ML	Mali
MN	Mongolia
MR	Mauritania
MW	Malawi
MX	Mexico
NE	Niger
NL	Netherlands
NO	Norway
NZ	New Zealand
PL	Poland
PT	Portugal
RO	Romania
RU	Russian Federation
SD	Sudan
SE	Sweden
SG	Singapore
SI	Slovenia
SK	Slovakia
SN	Senegal
SZ	Swaziland
TD	Chad
TG	Togo
TJ	Tajikistan
TM	Turkmenistan
TR	Turkey
TT	Trinidad and Tobago
UA	Ukraine
UG	Uganda
US	United States of America
UZ	Uzbekistan
VN	Viet Nam
YU	Yugoslavia
ZW	Zimbabwe

Description

METHOD FOR THE SIMPLE AND ENVIRONMENTALLY BENIGN PULPING OF NONWOOD FIBROUS MATERIALS

Technical Field

The present invention relates generally to a pulping process for nonwood materials. More particularly, this invention relates to a simple and environmentally benign process for chemi-mechanical pulping of kenaf, industrial hemp, and other nonwood fiber source materials to produce a papermaking pulp.

Background Art

It will appreciated by one having ordinary skill in the art that trees and other woody plants are not the only source of fibers for papermaking. There are a variety of nonwood annual and perennial plants which produce fibers having sufficient strength and length to produce paper with acceptable qualities. These nonwood plants are often referred to in that art as "agricultural residues" or "fiber crops". Examples of plants for each of these categories include:

Agricultural Residues	Fiber Crops
Wheat straw	Kenaf
Rice straw	Industrial hemp
Corn stalks	Sisal

Bagasse (sugar cane)	Rye grass
Flax straw	

One of the main advantages of these fiber sources is that they are perceived in the art as environmentally-benign alternatives to the use of trees. Indeed, nonwoods are currently the major source of papermaking fiber for developing countries and countries lacking significant wood resources.

For the most part, however, the development of a nonwood fiber industry in North America has been retarded due to the fact that nonwoods pulps are usually more expensive on a per-ton basis than wood pulps. Recently, several factors have dramatically increased the level of industry interest in these nonwood fiber sources. Some of these factors include environmental pressure to stop using trees; projections of world fiber shortage by 2010 and the need to find alternative fiber sources; abundance of agricultural residues (such as wheat straw) that are otherwise burned off fields; and opportunities to produce multiple products (oils, textile fibers, papermaking fibers, board fibers, plastics, food) from a simple fiber source, which provides unique opportunities for sustainable agriculture.

However, effective use of nonwood fiber sources presents some significant challenges that must be overcome. These challenges include the following:

(1) nonwoods must be harvested annually and stored, and thus, are sensitive to growing season, harvest conditions, etc.;

(2) nonwoods have a low bulk density compared to trees, and thus, can be hard to store and transport;

(3) nonwoods may require large amounts of herbicides and pesticides as compared to trees;

(4) nonwoods generally require smaller mills due to transport constraints, and it is often difficult to establish efficient chemical recovery systems for small mills; and

(5) nonwoods comprise fibers that may be shorter, more slender, or weaker than wood fibers.

One of the major areas of study in nonwoods is the development of so-called "mini-mill" processes. A suitable mini-mill would produce about 50-300 tons/day of fiber, as opposed to the 1000-2000 tons/day produced by modern "mega-mills." Although the larger mills are quite efficient, finding locations and obtaining environmental permits for them is becoming increasingly difficult. In addition, the ability to economically transport low-density nonwood fiber materials to a large mill is limited.

Thus, the main challenge in reducing nonwood raw materials into fibers for papermaking is to find an optimal pulping method for application in a mini-mill setting. The term "pulping" is generally defined as the reduction of the bulk fiber source material into its component fibers. The key is to perform this reduction without damaging the fiber (thereby reducing strength) or without losing too much fiber that will be suitable for papermaking (termed a "yield loss").

Several classes of pulping processes are generally known in the art. These processes include the following:

(1) Chemical Pulping -- In this type of pulping, a large chemical dose is used to dissolve away most of the lignin (glue) which holds the fibers together in the raw material. This dissolution is carried out in a digester, where chemicals are mixed with the raw material and then heated to medium to high temperature (100-170°C) and high pressure (2-15 atm). Standard digestion processes are carried out for about 1-8 hours. At the end of the digestion, the fibers are washed to separate them from the liquor, which contains dissolved lignin and spent chemicals. Elaborate systems have been developed to thicken and burn the liquor in order to recover heat energy from the lignin and regenerate the chemicals for use in subsequent digestion procedures.

Pulps from full chemical processes are characterized by high purity (high cellulose content, low hemicellulose and lignin content), suitable cleanliness levels, and suitable strength. With subsequent bleaching, high-brightness pulps for demanding printing and writing grade paper products may be produced. However, the processes often have a low yield (30-50%) due to chemical dissolution. In addition, full chemical processes require high capital investment and high operating costs. Thus, standard full chemical pulping processes are generally not suitable for nonwoods pulping applications.

(2) Mechanical Pulping -- In this type of pulping, raw materials are separated into fibers using brute mechanical force. Usually, the raw material is placed between rotating refiner plates, which shear it apart. Heat can be

applied to soften the fibers prior to refining. Yield from these types of processes is typically high (65-95%), but the quality of pulps is usually inferior to chemical pulps. Because there is still a large amount of lignin on the fiber surfaces, bonding sites are blocked, resulting in lower strength properties. Sheet flexibility is also reduced because lignin is left in the fiber walls. Overall, mechanical pulps are useful only for low-end paper grades like newsprint or catalog. However, since large quantities of chemicals are not required, chemical recovery is no problem. In addition, capital and operating costs are manageable. However, because of the limitation on pulp, and subsequently paper, quality described above, a purely mechanical process is also believed to have limited application to the pulping of nonwoods.

(3) Chemi-Mechanical Pulping -- This type of pulping uses aspects of both of the previously described process types. Raw material is impregnated with small amounts of chemicals to soften the lignin, and then it is mechanically treated to complete the separation. Heat is typically applied to improve pulping. With this hybrid process, good fiber properties may be developed without extensive chemical application. In addition, capital and operating costs are almost as low as for pure mechanical pulping. Pulps from chemi-mechanical processes can be used for low- to medium- quality papers, and with additional processing they may be used for some high-end purposes. However, a chemi-mechanical pulping process suitable for the pulping of nonwoods has not been described in the art.

Prior art patent references of interest include U.S. Patent Nos. 4,756,799 and 4,900,399, both to Bengtsson et al., describing methods for manufacturing bleached chemi-mechanical and semi-mechanical fiber pulp by means of a one or two stage impregnation process. However, these patents particularly describe production of a pulp from wood materials. The described methods particularly require that the wood material be preheated before it is subjected to mechanical manipulation in a standard twin disk refiner. Thus, the method steps described in these patents are not believed to have particular application to a pulping process for nonwood materials.

U.S. Patent Nos. 4,997,488 to Gould et al.; 4,806,475 to Gould; 4,774,098 to Gould et al.; and 4,649,113 to Gould describe alkaline peroxide treatment of nonwoody lignocellulosics and products made by such treatments. However, the primary focus of these patents is the production of nutritional supplements, culture media or other compounds from cellulose in the nonwoody materials for use in feeding domestic animals, humans, or in the growth of microbial cultures. Stated differently, the focus of the methods described in these patents is to produce materials from cellulose that can be metabolized by animals.

Heretofore, there has been no suggestion in the art of a suitable application of any pulping process to nonwood fiber source materials. Indeed, for nonwoods, the most sensible approach is to install a small mill at the center of a defined growing area. This mill should use a simple process, with low operating and capital costs, to maintain economies of scale equal to those of

the mega-mill. The process should render the mill almost invisible to the environment, and should require only small amounts of environmentally-benign chemical agents. Such a process is not currently available in the art.

Summary of the Invention

A process for producing a pulp suitable for use in papermaking from a nonwood fiber source material has been discovered by the applicant. The process comprises the steps of washing the nonwood fiber source material with a washing solution; impregnating the nonwood fiber source material with a pulping solution at ambient temperature and at atmospheric pressure; and mechanically shearing the nonwood fiber source material to form a papermaking pulp. Preferably, the process further comprises compressing the nonwood fiber source material to form a compressed plug after washing the nonwood fiber source material and prior to impregnating the nonwood fiber source material with the pulping solution.

Accordingly, it is an object of this invention to provide a nonwoods pulping process that uses whole-stalk, chopped material directly from the field with no need for a separation step or other pre-processing prior to introduction into the process.

It is a further object of this invention to provide a nonwoods pulping process wherein the fiber from the field is washed and cleaned as a part of the process with no requirement for separate equipment for washing and cleaning.

It is another object of this invention to provide a nonwoods pulping process wherein dirt, sand, fine fibers, and core material are expelled from the process before pulping, resulting in a cleaner final pulp and in more efficient use of chemicals.

It is another object of this invention to provide a nonwoods pulping process which occurs at room temperature and wherein no heating or steaming is required.

It is another object of this invention to provide a nonwoods pulping process which occurs at atmospheric conditions, thereby eliminating the need for a pressure vessel in the system.

It is another object of this invention to provide a flexible nonwoods pulping process wherein a range of pulp qualities may be produced, depending on chemical addition and refining intensity.

It is another object of this invention to provide a nonwoods pulping process wherein the primary portion of the process can occur over a short period of time, such as thirty minutes, so that vessel sizes are manageable.

It is another object of this invention to provide a pulp with high freeness characteristics.

It is still another object of this invention to provide a nonwoods pulping process that uses readily available and inexpensive equipment.

Some of the objects of the invention having been stated hereinabove, other objects will become evident as the description proceeds, when taken in connection with the accompanying drawings as best described hereinbelow.

Brief Description of the Drawings

Figure 1 is a schematic representation of the primary nonwoods pulping process of this invention;

Figure 2 is a schematic representation of the nonwoods pulping process of this invention wherein secondary refining of the pulp by treatment with acidified water is depicted;

Figure 3 is a schematic representation of the nonwoods pulping process of this invention wherein secondary refining of the pulp with a bleaching solution is depicted; and

Figure 4 is a schematic representation of the nonwoods pulping process of this invention wherein secondary refining of the pulp by treatment with acidified water/chelant and by a bleaching solution is depicted.

Detailed Description of the Invention

The novel process of the instant invention addresses the paper industry's need for a mini-mill process for use with nonwood fibers. This process is a chemi-mechanical process, using small amounts of chemicals and moderate mechanical action to produce fair- to high-quality pulps.

The term "woody" is used herein both in the botanical sense to mean "comprising wood"; that is, composed of extensive xylem tissue as found in trees and shrubs, and also in the sense of being "wood-like". Accordingly, the terms "nonwood", "nonwoods", and "nonwoody" refer to materials lacking these characteristics.

An excellent candidate for a source of nonwood fiber material is kenaf, a hearty annual plant of the hibiscus family. Kenaf can be a grown in almost every state of the United States. In addition to kenaf, another candidate is industrial hemp, the low-THC relative of marijuana. Grown extensively in North America in the early part of this century, hemp was arbitrarily outlawed by the government due to its connection with marijuana. However, there is a groundswell of support to legalize industrial hemp, due to its outstanding fiber properties and high yield. Additionally, industrial hemp is easy to grow requiring no pesticides or herbicides. Thus, there is good reason to believe that it will be legalized in the near future. Other candidate agricultural residues and fiber crops include wheat straw, rice straw, corn stalks, bagasse (sugar cane), flax straw, sisal, and rye grass.

Primary Pulping

Stalks of material (e.g. kenaf, hemp) are harvested whole and chopped into pieces 2-4 inches in length (this dimension will depend on the type of feed screw selected). Both core and bark are used, without separation. Separated material may be used, if desired, to get more specified properties.

Referring now to the drawings, where like reference numerals refer to like parts throughout, the process of this invention is described schematically. Referring particularly to Figure 1, chopped raw material is fed into the base of a conventional, twin-flight, incline washing screw **12**. As the material moves upward in screw **12**, it is wetted and washed by the counter-current flow of the process wash/filtrate, which is introduced at the top **14** of the screw **12**. The

filtrate is recycled from the sump **18** at the bottom **16** of screw **12**. At steady state, a small side stream **20** from sump **18** is bled off and sent either to a water treatment system or to some other non-process use. It is preferred that accumulated dirt and metal ions be removed from stream **20**. Fresh or treated water stream **22** is then introduced into the process to compensate for the loss in stream **20**.

Optionally, depending on the type of fiber used and the degree of final pulp brightness desired, a steady-state concentration of a commercially-available chelating agent, such as diethylene-triamine-pentaacetic acid (DTPA), ethylene-diamine-tetraacetic acid (EDTA), can be maintained in the wash filtrate. The experiments described in the Examples presented below were performed using Vinings Industries VINKEEL^{tm} 2010 brand chelating agent, a lower-cost variation of these agents.

The chelating agent sequesters and ties up metal ions, such as iron, manganese, calcium, and cobalt, which tend to decompose hydrogen peroxide and limit brightness development during pulping. In addition, the chelating agent provides for improved washing of the fiber. Depending on fiber type and condition, chelation can be aided by the use of acid to reduce the pH of the wash filtrate to pH 5-6. However, such acidification will result in higher alkali demand in the pulping stage, and it may affect brightness development during pulping. Thus, care should be taken in the use of acid.

Additionally, depending on fiber type and pulp brightness, steam or heating can be added to the wash filtrate system to improve washing and

chelation of metals. However, care should be used to avoid heating the raw material to the point that it causes alkaline darkening during the pulping stage. Indeed, prior art alkaline peroxide pulping or bleaching processes use high temperatures (50-110°C) to take advantage of significantly increased reaction rates. However, a competing reaction occurs at these temperatures, in which alkali causes formation of color-causing ("chromophoric") groups, which lead to a loss of brightness in the pulp. As a result of this alkaline darkening, additionally bleaching is usually required in prior art processes. In the process of the instant invention, alkaline darkening is avoided by utilizing lower reaction temperatures. Thus, in the process of the instant invention, the brightening obtained per unit of peroxide consumed is quite high.

Filtrate bled off from the sump **18** will contain dirt, silt, sand, fine fiber particles, and dissolved metal ions from the raw material **10**. If no chelant is used, the waste material is considered completely organic and chemical-free. Thus, it can be recycled back to the farm or to other land-spread uses with little environmental concerns.

Though preferred, washing screw **12** is not essential to the process of this invention. Any method of thoroughly wetting the raw material **10** prior to passing it through the impregnation screw may be acceptable. However, for some products, removal of fine contaminants (e.g. sand), plant extractives, and metal ions (via chelation) will be important. Additionally, the filtrate from the wash may contain valuable plant components that can be salvaged.

Referring again to Figure 1, nonwood fiber source material from washing screw **12** falls into infeed hopper **24**. The housing **26** of infeed hopper **24** is perforated to allow free filtrate to escape from the washed material. The filtrate then flows into sump **18**. Optionally, a short drainage conveyor **28** may be placed prior to the hopper **24** to facilitate drainage.

An infeed screw **30** inside the hopper **24** then transports the washed material into impregnation screw **32**. Impregnation screw **32** is manufactured so that the diameter of the overall screw remains the same while the shaft diameter increases and the open flight area decreases. The effect of screw **32** on washed material is to compress it, reducing volume to one fourth or less of original volume.

A housing **34** surrounds screw **32**. Housing **34** is perforated so that as material is intensely squeezed, free wash water, dirt, silt, sand, crushed core material, fines, and trash are expelled from it. This compression step completes the thorough washing phase of the process.

Waste material expelled from impregnation screw **32** is in the form of a thick sludge. If neither chelating agent nor acid has been used during the washing stage, the waste material may be further thickened and recycled to the farm land or to other land-spread applications with little or no environmental concerns.

Impregnation screw **32** also exerts an initial mechanical action on the nonwood fiber source material. Long bast fibers within the material are twisted and compressed, and may be shortened somewhat. Core material within

material is crushed into small, splinter-like pieces that are easier to impregnate with chemicals.

At the end **36** of impregnation screw **32** a compressed plug of cleaned nonwood fiber source material is formed. The plug is pushed by screw **32** into impregnation vessel **38**, a small box just inside of a larger reaction vessel **40**. Vessel **38** is kept filled with chemical pulping solution. By the term "pulping solution" it is meant any solution capable of breaking down lignin in the fibers of the nonwood material, causing the fibers to swell, proving proper pH for bleaching, providing for bleaching itself, stabilizing the bleaching reactions, and/or accomplishing any other reaction associated with preparing a papermaking pulp as would be apparent to one having ordinary skill in the art.

When the plug emerges into vessel **38**, it expands and absorbs the pulping solution deeply. Impregnated material is lifted out of vessel **38** by twin screws **42**, to a small area above the fill line of vessel **38** where the material drains briefly. After draining, the material overflows into the outer reaction vessel **40**. The level of material is maintained in reaction vessel **40** so that the total residence time within vessel **40** is 20-30 minutes. Longer residence times may be used to further brighten the nonwood fiber source material, but 30 minutes has been found to be an optimal residence time.

The chemical agents used in the pulping solution preferably comprise sodium hydroxide, hydrogen peroxide, and magnesium carbonate. Sodium hydroxide serves as a source of alkali, which breaks down lignin in the fibers, causes the fibers to swell, and provides the proper pH for peroxide bleaching.

Hydrogen peroxide, under alkaline conditions, provides for some degradation of lignin. However, hydrogen peroxide primarily reacts with color-causing groups (chromophores) in the fiber, breaking them down so that they can no longer impart dark color to the pulp. Magnesium carbonate is used as a source of magnesium ions, which have been found to stabilize reactions of alkaline hydrogen peroxide. At the lower temperatures utilized in the process of this invention, the effectiveness of magnesium carbonate and similar compounds is not as great as at higher temperatures. Therefore, use of magnesium carbonate and similar compounds is optional, depending on the fiber type and the number and type of metal ions present in the water system being used.

In addition, small amounts of the same chelating agent used in the wash stage may be added. Depending on the metal ion content of the raw material, addition of chelant in the pulping solution may increase brightness of the final pulp. Finally, other sources of alkali and peroxide would be well known to one having ordinary skill in the art. Particular examples include potassium hydroxide and sodium peroxide.

Thus, at a minimum, the pulping solution preferably comprises: (a) some source of alkali, which is necessary for the swelling and softening of the raw material, for partially reacting with some of the lignin in the raw material, and for establishing proper conditions for peroxide brightening; and (b) some source of peroxide, which is an effective brightening agent under the conditions of the process of the instant invention. Additionally, the pulping solution can

comprise small quantities of metal sequestering and chelating agents, such as magnesium compounds or VINKEEL^{tm} 2000.

All chemical agents are prepared in aqueous solution at a concentration sufficient to deliver the proper dosage of chemical. This concentration will vary depending upon the absorbtivity of the raw material. Thus, if a material picks up a large amount of liquid during impregnation, chemical agents may be prepared at a lower solution concentration than if the material picks up smaller amounts of liquid. Given the preferred shorter residence time in vessel **40** as described above, relatively small volumes of pulping solution are required, as compared to prior art processes. Finally, all impregnation and reaction takes place at atmospheric conditions and at ambient temperature. The preferred temperature thus ranges from about 20°C to about 40°C.

Continuing with Figure 1, a discharge screw **44** is located at the bottom of the reaction vessel **40** and removes material continuously, with speed controlled so as to maintain the desired level of nonwood fiber source material in reaction vessel **40**. Screw **44** feeds material into a refiner feed screw **46**, which in turn feeds the material into the eye of a twin-disk refiner **48**. A suitable example of refiner **48** is a SUNDS DEFIBRATOR CD-300^{tm}, with 12-inch plates. A 300-HP motor rotates one plate **50** at 3600 RPM, while the other plate **52** is stationary. The shaft driving the rotating plate **50** is movable, so that the gap between plates **50** and **52** may be reduced to increase refining intensity or increased to reduce refining intensity. Refining intensity is controlled by monitoring the load on the motor, and this load is translated into

the specific energy input into the nonwood fiber source material. Notably, there is no need to heat the nonwood fiber source material prior to introduction into refiner **48**.

When the impregnated and reacted material is introduced between refiner plates **50** and **52**, it is sheared across the surfaces of the two plates **50** and **52**, causing the bast and core fibers to undergo intense mechanical action. Long bast macrofibers are separated into smaller unit fibers, while the core pieces are both pulverized and separated into fibers.

Centrifugal force causes the refined nonwood fiber source material to be flung out of refiner **48** through a bottom port **54**. The material at this point comprises a papermaking pulp, with a fluffy, spongy feel and a bright yellow appearance. Friction in refiner **48** can cause the pulp to be heated up to 50-60°C at the discharge. Residual alkali typically gives the pulp a pH of about 9.

All the equipment described herein is well-known in the art and is available commercially from a variety of manufactures, including for example, Sunds Defibrator Co., of Norcross, Georgia.

<u>Secondary Refining</u>

Pulp quality can be optimized by using the primary pulping stage described above to grossly defiber the nonwood fiber source material, followed by a second mechanical refining stage to reduce shives and develop strength properties.

Referring now to Fig. 2, pulp discharging from primary refiner **48** falls into a transfer conveyor **56**, which takes the pulp into a feed chute **58** for a secondary refiner **60**, which is identical to the primary refiner **48**, and includes plates **62** and **64**. From the chute **58**, another feed screw **66** introduces the fiber into the secondary refiner **60**. Plate gap and specific energy input are adjusted to yield desired pulp properties.

Other Secondary Treatment

Treatment of the pulp after secondary mechanical refining will depend upon the brightness desired for the final pulp. Three examples are described hereinbelow, which are shown schematically in Figures 2-4. The examples are provided to illustrate the invention described herein, and should not be construed as limiting the scope of applicant's invention.

Example 1: Lower Brightness (55-60 ISO)

This brightness range is generally suitable for low-end products like cover stock, dyed stock, art papers, etc. In this case, the brightness of the pulp emerging from the refiners is adequate, and no further bleaching is necessary. However, the residual alkali in the pulp must either be washed out or neutralized before the pulp can be processed into paper. The unit ISO is widely used in the art to characterize brightness, and refers particularly to the reflectance of a sheet of paper compared to a tile of magnesium oxide using an incident light source with a wavelength of 457 nm. White and bright printing and writing papers are usually in the 80-90% ISO brightness range, while newsprint and lower-brightness papers are in the 55-70% brightness range.

Washing of the pulp is not necessary after refining. Instead, the pulp is mixed with sulfuric acid until a pH of 7-7.5 is achieved, thereby neutralizing the residual alkali. The color of the pulp will turn from yellowish to whitish during this treatment.

Referring again to Fig. 2, neutralization is accomplished by injecting acidified dilution water **68** into the flat zone of the secondary refiner plates **62** and **64**. In normal refiner operation, a small quantity of dilution water is injected into the refiner to facilitate refining action and also to prevent pulp burning from intense friction. Rotation of the refiner plates **62** and **64** provides an ideal mixing zone, so the addition of acidified water **68** to the refiner **60** results in thorough mixing of acid with the pulp. Additional acidified water **70** may be added to the pulp as it discharges from the refiner.

To provide adequate time for the acid to react with the pulp, the pulp is allowed to fall into a standpipe **72** where it remains for preferably about 15 minutes. The concentration of acid in the dilution water **68** is adjusted so that the pH of the pulp at the bottom of standpipe **72** is 7-7.5.

After neutralization, the pulp may be discharged from the standpipe **72** using a screw or pump. The pulp is now ready for optional final refining/beating, screening/cleaning and papermaking, or thickening/drying for storage.

<u>Example 2: Moderate Brightness (60-70 ISO)</u>

This brightness range is suitable for newsprint and some illustrative grades. To achieve the extra brightness, it is necessary to perform some post-

refining bleaching. However, the brightness gain can be achieved without the pulp neutralization, chelation, or washing steps found in complex, expensive and possibly environmentally harmful prior art processes.

Referring now to Fig. 3, a solution **74** made up of sodium hydroxide, hydrogen peroxide, magnesium carbonate, and a chelating agent is prepared. Sodium silicates, which tend to stabilize peroxide compounds, may also be added. It is also possible to use the same solution as is used for impregnation/pulping as described above. Solution **74** is injected into secondary refiner **60** as dilution water between plates **62** and **64**. Thus, the added chemicals are thoroughly mixed with the pulp. Pulp pH should be between 11-12.

The pulp falls into the standpipe **72**, which has adequate volume to provide a residence time of 30 minutes. At the bottom of standpipe **72**, the pulp pH and residual peroxide concentration are monitored, and chemical addition and/or concentration is modified to provide the desired readings at this point. The peroxide bleaching reaction should take place at 80-90°C. Heating for this reaction is accomplished by heating the injected chemical solution and/or injecting steam into refiner **60**.

From the bottom of the standpipe **72**, the pulp is pumped or otherwise transferred into the bottom **78** of a standard upflow reaction tower **76**. Tower **76** provides a volume sufficient for a residence time of three hours. The pulp rises and is removed at the top **80** of the tower **76** using a standard discharge device.

Since more alkali has been added to the pulp for peroxide bleaching, the use of acid neutralization will usually be too costly. It is thus optionally necessary to feed the pulp into a standard washing device **82**. After washing, the pulp is ready for final refining/beating, screening, cleaning, papermaking, etc.

Example 3: Higher Brightness (70 ISO and above)

For some publication grades and higher-end uses, higher final pulp brightness is required. This brightness range may be accomplished using the process setup described in Example 2. However, depending upon the type of raw material and its metals content, it can be necessary or desirable to perform an additional acid chelation step followed by a bleaching step.

Referring now to Fig. 4, chelation is accomplished by injecting chelating solution **84** into refiner **60** instead of dilution water. Chelating solution **84** includes enough acid to reduce pulp pH to 5-6 after the pulp has remained in standpipe **72** for approximately 30 minutes. In addition, solution **84** contains a small quantity of chelating agent, similar to that used for impregnation. Standpipe **72** provides volume sufficient for a residence time of 30 minutes. At the bottom of standpipe **72**, the pulp is transferred into a standard thickening device **86**, such as a screw or belt press. A washer may also be used at this point, but it has been observed that washing is not required to achieve higher brightness level. If utilized, a washing step will reduce the amount of alkali required in the peroxide bleaching stage.

After discharge from thickening device **86**, the pulp falls into a standard chemical mixer **88**. The pulp is mixed with a solution **90** comprising sodium hydroxide, hydrogen peroxide, magnesium carbonate, and a small amount of chelating agent. Sodium silicates, which tend to stabilize peroxide compounds, may also be added. The solution used for impregnation is a suitable example. The pulp is then introduced into a standard steam mixer **92**, where it is heated to 80-90°C. The heated pulp is then introduced into the bottom of upflow tower **76**. At the top **80** of the tower **76**, the pulp is washed prior to subsequent processing.

<u>Representative Process Specifications for Examples 1-3</u>

By way of example, using a single refiner and the optimal chemical dosages specified hereinbelow, plus an additional bleaching stage, a pulp was produced from whole-stalk kenaf with brightness of 65 ISO (sufficient for newsprint). The cleanliness of the pulp was outstanding with respect to dirt, sand, and fines, even though no auxiliary cleaning was performed. Sheet strength properties were tested and found to be similar to those from full chemi-thermo-mechanical processes in the prior art. Similar results were obtained using whole-stalk industrial hemp.

The preferred process specifications for Examples 1-3 are as follows:

(1) Wash filtrate temperature: system steady state temperature

(2) Range of impregnation chemical dosage (as wt. % on OD fiber):

Sodium hydroxide	2-6%
Hydrogen peroxide	2-6%

Magnesium carbonate	0.5 %
Chelant	depends on agent; 0.2 - 0.5% for VINKEEL^{tm} 2010

(3) Determined optimal dosages for impregnation (as wt. % on OD fiber):

Sodium hydroxide	2 %
Hydrogen peroxide	2 %
Magnesium carbonate	0.5%
Chelant (VINKEEL^{tm} 2010)	0.2%

(4) Impregnation temperature: ambient (preferably ranging from 20-40°C)

(5) Reaction time: 20-30 minutes

(6) Volume of reaction vessel: sufficient to provide 20-30 minutes reaction time

(7) Reaction temperature: ambient (preferably ranging from 20-40°C)

(8) Reaction consistency: approximately 14 %

(9) Primary refiner gap: 0.3 - 0.35 mm

(10) Primary refiner specific energy input: 300-600 kwh/MT

(11) Primary refiner dilution flow: 0.55 gpm

(12) Pulp consistency after first refiner: approximately 10 %

(13) Pulp pH after first refiner: approximately 9

(14) Pulp freeness after first refiner: approximately 350-400 ml CSF

(15) Secondary refiner plate gap: 0.25-0.4 mm

(16) Secondary refiner specific energy input: depends on pulp properties -- 200-400 kwh/MT

(17) Pulp consistency after secondary refiner: 9-10 %

(18) Pulp pH after secondary refiner: approximately 9 pH

(19) Pulp brightness after secondary refining, neutralization: approximately 55 ISO

(20) Pulp freeness after secondary refining, neutralization: approximately 250-350 ml CSF

(21) Fiber yield after secondary refining: approximately 65%

The term "consistency", as used above in referring to "reaction consistency" and to "pulp consistency", is used in the art to denote percent (%) solids of the pulp slurry.

The term "freeness", as used above in referring to "pulp freeness", refers to the drainage rate of pulp, or how "freely" the pulp will give up its water. Freeness is important in papermaking in that, if the freeness is too low, it is not possible to remove enough water on the paper machine to achieve good sheet structure and strength. Often, mechanical pulps have low freeness due to harsh action imparted to the raw material, which produces fines and particles which plug up the draining paper mat. Many chemical pulping process pulping processes using whole-stalk materials (both bast and core) for kenaf and industrial hemp have problems with poor freeness, due to over-pulping of the core fraction.

The process of the instant invention does not suffer from the freeness problems of prior art processes. Indeed, the process of the instant invention produces a mechanical pulp with high freeness. Particularly, for the instant process, pulp freeness after first refiner ranges from approximately 350-400 ml CSF. Further, pulp freeness after secondary refining and neutralization ranges from approximately 250-350 ml CSF. Accordingly, the term "high freeness" is meant to refer to a freeness of about 250 ml CSF and above.

Thus, important aspects of the instant invention include the use of whole-stalk, unseparated raw materials without preprocessing. Further, the process of this invention incorporates washing, cleaning and metals removal (chelation) into simple and readily available equipment. The final pulp is adequately cleaned for many applications without additional screening and/or cleaning, a characteristic previously undescribed in the art for whole-stalk field materials. Moreover, the process of this invention uses only small quantities of chemicals, roughly one-tenth of the quantities used for standard full chemical pulping, to produce a pulp with acceptable strength and brightness.

Significantly, the process of this invention provides for the impregnation, reaction and refining of the pulp without auxiliary heating. Stated differently, the process is preferably performed at ambient temperature. Prior art processes use alkaline peroxide at elevated temperatures (60°C and above). The peroxide brightening reaction is competing with a simultaneous alkaline darkening reaction. By using peroxide at ambient temperature as described in the process of applicant's invention, an efficient brightening is achieved with

a small chemical dosage. Additionally, the impregnation, reaction with pulping solution, and refining of the nonwood fiber source material in the process of this invention occurs at atmospheric pressure, thereby simplifying the overall process by elimination of complex and expensive pressure vessels.

As described in Example 1, the process of this invention produces a pulp that is ready for papermaking with simple neutralization and no washing, wherein the neutralization of the pulp by injecting acidified water into the refiner makes additional chemical mixtures unnecessary. As described in Example 2, the instant process also provides for the production of pulp that is ready for papermaking using simple post-refining bleaching by injecting bleaching agents into the refiner, making additional chemical mixers unnecessary. As set forth in Example 3, the instant process provides for the production of a pulp that is ready for papermaking using a neutralization/chelation prior to subsequent bleaching, by injecting acidified water in chelant into the refiner.

Thus, the instant process provides for the production of a good quality pulp at relatively high yield (65% of raw material recovered as useable pulp) and with high freeness characteristics, without the use of high temperatures and pressures. The instant process is a full pulping and bleaching process that produces a minimal volume of effluent, eliminating the need for complex chemical recovery systems. Effluent from acid neutralization of the pulps after refining possesses environmentally benign characteristics and are easily treated with simple aerated lagoons. Finally, the instant process provides for the production of papermaking pulps from nonwood materials using minimal

capital equipment and operating costs, thereby expanding the availability of nonwood materials for use in papermaking.

It will be understood that various details of the invention may be changed without departing from the scope of the invention. Furthermore, the foregoing description is for the purpose of illustration only, and not for the purpose of limitation--the invention being defined by the claims.

CLAIMS

What is claimed is:

1. A process for producing a pulp suitable for making paper from a nonwood fiber source material, the process comprising the steps of:

(a) washing a nonwood fiber source material with a washing solution;

(b) impregnating the nonwood fiber source material with a pulping solution at substantially ambient temperature and at atmospheric pressure;

(c) feeding the impregnated nonwood fiber source material into a mechanical shearing system at ambient temperature; and

(d) mechanically shearing the nonwood fiber source material to form a papermaking pulp.

2. The process of claim 1, wherein the washing solution of step (a) comprises a chelating agent.

3. The process of claim 1, further comprising mechanically mixing the washing solution with the nonwood fiber source material in step (a).

4. The process of claim 1, further comprising heating the process during step (a) to a temperature above ambient temperature but below a temperature at which alkaline darkening occurs.

5. The process of claim 1, wherein the washing solution has a pH ranging from about 5 to about 6.

6. The process of claim 1, wherein said impregnating the nonwood fiber source material occurs at a temperature ranging from about 20°C to about 40°C.

7. The process of claim 1, wherein said pulping solution comprises an alkaline solution.

8. The process of claim 7, wherein said pulping solution comprises an alkaline peroxide solution.

9. The process of claim 8, wherein said pulping solution comprises sodium hydroxide, hydrogen peroxide and magnesium carbonate.

10. The process of claim 8, further comprising the step of neutralizing residual alkali in the papermaking pulp by addition of an acid solution.

11. The process of claim 10, further comprising the step of mechanically shearing the papermaking pulp while adding the acid solution.

12. The process of claim 1, wherein the nonwood fiber source material is selected from the group consisting of kenaf, industrial hemp, sisal, rye grass, wheat straw, rye straw, corn stalks, bagasse and flax straw.

13. The process of claim 1, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 250 ml CSF at the lowest.

14. The process of claim 13, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 350 ml CSF at the lowest.

15. The process of claim 1, further comprising the step of compressing the nonwood fiber source material to form a compressed plug after washing the nonwood fiber source material but prior to impregnating the nonwood fiber source material with a pulping solution.

16. The process of claim 1, further comprising the steps of:

(a) washing the papermaking pulp with an acid solution including a chelating agent;

(b) thickening the papermaking pulp;

(c) treating the papermaking pulp with an alkaline bleaching solution at a temperature ranging from about 80°C to about 90°C; and

(d) washing the papermaking pulp.

17. The process of claim 1, further comprising the step of treating the papermaking pulp with a bleaching solution at a temperature ranging from about 80°C to about 90°C.

18. The process of claim 17, further comprising washing the papermaking pulp after the treating with the bleaching solution.

19. The process of claim 17, further comprising mechanically shearing the papermaking pulp during the treating with the bleaching solution.

20. The process of claim 19, further comprising mechanically shearing the pulp to a freeness of about 250 ml CSF at the lowest.

21. The process of claim 20, further comprising mechanically shearing the pulp to a freeness of about 350 ml CSF at the lowest.

22. A process for producing a pulp suitable for papermaking from a nonwood fiber source material, the process comprising the steps of:

(a) washing a nonwood fiber source material with a washing solution;

(b) compressing the nonwood fiber source material to form a compressed plug;

(c) impregnating the compressed plug of nonwood fiber source material with a pulping solution at a temperature ranging from about 20°C to about 40°C and at atmospheric pressure;

(d) feeding the impregnated nonwood fiber source material into a mechanical shearing system at ambient temperature; and

(e) mechanically shearing the compressed plug of nonwood fiber source material to form a papermaking pulp.

23. The process of claim 22, wherein the washing solution of step (a) comprises a chelating agent.

24. The process of claim 22, further comprising mechanically mixing the washing solution with the nonwood fiber source material.

25. The process of claim 22, further comprising heating the washing solution during step (a) to a temperature above ambient temperature but below a temperature at which alkaline darkening occurs.

26. The process of claim 23, wherein the washing solution has a pH ranging from about 5 to about 6.

27. The process of claim 22, wherein the pulping solution comprises an alkaline solution.

28. The process of claim 22, wherein the pulping solution comprises an alkaline peroxide solution.

29. The process of claim 28, wherein the pulping solution comprises sodium hydroxide, hydrogen peroxide, and magnesium carbonate.

30. The process of claim 28, further comprising neutralizing residual alkali in the papermaking pulp by addition of an acid solution.

31. The process of claim 30, further comprising mechanically shearing the papermaking pulp while adding the acid solution.

32. The process of claim 22, wherein the nonwood fiber source material is selected from the group consisting of kenaf, industrial hemp, sisal, rye grass, wheat straw, rye straw, corn stalks, bagasse and flax straw.

33. The process of claim 22, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 250 ml CSF at the lowest.

34. The process of claim 33, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 350 ml CSF at the lowest.

35. The process of claim 22, further comprising the steps of:

(a) washing the papermaking pulp with an acid solution including a chelating agent;

(b) thickening the papermaking pulp;

(c) treating the papermaking pulp with an alkaline bleaching solution at a temperature ranging from about 80°C to about 90°C; and

(d) washing the papermaking pulp.

36. The process of claim 22, further comprising treating the papermaking pulp with a bleaching solution at a temperature ranging from about 80°C to about 90°C.

37. The process of claim 36, further comprising washing the papermaking pulp after treating with the bleaching solution.

38. The process of claim 36, further comprising mechanically shearing the papermaking pulp during the treating with the bleaching solution.

39. The process of claim 38, further comprising mechanically shearing the pulp to a freeness of about 250 ml CSF at the lowest.

40. The process of claim 39, further comprising mechanically shearing the pulp to a freeness of about 350 ml CSF at the lowest.

AMENDED CLAIMS

[received by the International Bureau on 28 June 1999 (28.06.99); original claims 20,24,33 and 39 cancelled; original claims 1-4,8,10-11, 14,16,19,21-23,25,28,30,31,34-35 and 40 amended; new claims 41-44 added, remaining claims unchanged (7 pages)]

1. A process for producing a pulp suitable for making paper from a nonwood fiber source material, the process comprising the steps of:

(a) providing a nonwood fiber source material comprising bast and core fibers;

(b) washing the nonwood fiber source material with a washing solution;

(c) impregnating the nonwood fiber source material with a pulping solution at substantially ambient temperature and at atmospheric pressure;

(d) feeding the impregnated nonwood fiber source material into a mechanical shearing system at ambient temperature; and

(e) mechanically shearing the nonwood fiber source material to form a papermaking pulp.

2. The process of claim 1, wherein the washing solution of step (b) comprises a chelating agent.

3. The process of claim 1, further comprising mechanically mixing the washing solution with the nonwood fiber source material in step (b).

4. The process of claim 1, further comprising heating the process during step (b) to a temperature above ambient temperature but below a temperature at which alkaline darkening occurs.

5. The process of claim 1, wherein the washing solution has a pH ranging from about 5 to about 6.

6. The process of claim 1, wherein said impregnating the nonwood fiber source material occurs at a temperature ranging from about 20°C to about 40°C.

7. The process of claim 1, wherein said pulping solution comprises an alkaline solution.

8. The process of claim 7, wherein said pulping solution comprises an alkaline peroxide solution, wherein the peroxide is present in a dosage of about 2% weight on OD fiber.

9. The process of claim 8, wherein said pulping solution comprises sodium hydroxide, hydrogen peroxide and magnesium carbonate.

10. The process of claim 7, further comprising the step of neutralizing residual alkali in the papermaking pulp by addition of an acid solution.

11. The process of claim 10, further comprising the step of mechanically shearing the papermaking pulp while adding the acid solution to form a papermaking pulp having a brightness of at least about 55% ISO.

12. The process of claim 1, wherein the nonwood fiber source material is selected from the group consisting of kenaf, industrial hemp, sisal, rye grass, wheat straw, rye straw, corn stalks, bagasse and flax straw.

13. The process of claim 1, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 250 ml CSF at the lowest.

14. The process of claim 1, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 350 ml CSF at the lowest.

15. The process of claim 1, further comprising the step of compressing the nonwood fiber source material to form a compressed plug after washing the nonwood fiber source material but prior to impregnating the nonwood fiber source material with a pulping solution.

16. The process of claim 1, further comprising the steps of:

(a) washing the papermaking pulp with an acid solution including a chelating agent;

(b) thickening the papermaking pulp;

(c) treating the papermaking pulp with an alkaline bleaching solution while mechanically shearing the papermaking pulp;

(d) continuing treating the papermaking pulp with the alkaline bleaching solution at a temperature ranging from about 80°C to about 90°C; and

(e) washing the papermaking pulp to form a papermaking pulp having a brightness of at least about 65% ISO.

17. The process of claim 1, further comprising the step of treating the papermaking pulp with a bleaching solution at a temperature ranging from about 80°C to about 90°C.

18. The process of claim 17, further comprising washing the papermaking pulp after the treating with the bleaching solution.

19. The process of claim 17, further comprising mechanically shearing the papermaking pulp during the treating with the bleaching solution to form a papermaking pulp having a brightness of at least about 65% ISO.

20. (Canceled).

21. The process of claim 19, further comprising mechanically shearing the pulp to a freeness of about 350 ml CSF at the lowest.

22. A process for producing a pulp suitable for papermaking from a nonwood fiber source material, the process comprising the steps of:

(a) providing a nonwood fiber source material comprising bast and core fibers;

(b) washing and mechanically mixing the nonwood fiber source material with a washing solution;

(c) compressing the nonwood fiber source material to form a compressed plug;

(d) impregnating the compressed plug of nonwood fiber source material with a pulping solution at a temperature ranging from about 20°C to about 40°C and at atmospheric pressure;

(e) feeding the impregnated nonwood fiber source material into a mechanical shearing system at ambient temperature; and

(f) mechanically shearing the nonwood fiber source material to form a papermaking pulp having a freeness of at least about 250 ml CSF.

23. The process of claim 22, wherein the washing solution of step (b) comprises a chelating agent.

24. (Canceled).

25. The process of claim 22, further comprising heating the washing solution during step (b) to a temperature above ambient temperature but below a temperature at which alkaline darkening occurs.

26. The process of claim 23, wherein the washing solution has a pH ranging from about 5 to about 6.

27. The process of claim 22, wherein the pulping solution comprises an alkaline solution.

28. The process of claim 22, wherein the pulping solution comprises an alkaline peroxide solution, wherein the peroxide is present in a dosage of about 2% weight on OD fiber.

29. The process of claim 28, wherein the pulping solution comprises sodium hydroxide, hydrogen peroxide, and magnesium carbonate.

30. The process of claim 27, further comprising neutralizing residual alkali in the papermaking pulp by addition of an acid solution.

31. The process of claim 30, further comprising mechanically shearing the papermaking pulp while adding the acid solution to form a papermaking pulp having a brightness of at least about 55% ISO.

32. The process of claim 22, wherein the nonwood fiber source material is selected from the group consisting of kenaf, industrial hemp, sisal, rye grass, wheat straw, rye straw, com stalks, bagasse and flax straw.

33. (Canceled).

34. The process of claim 22, further comprising mechanically shearing the non-wood fiber source material to form a papermaking pulp having a freeness of about 350 ml CSF at the lowest.

35. The process of claim 22, further comprising the steps of:

(a) washing the papermaking pulp with an acid solution including a chelating agent;

(b) thickening the papermaking pulp;

(c) treating the papermaking pulp with an alkaline bleaching solution while mechanically shearing the papermaking pulp;

(d) continuing treating the papermaking pulp with the alkaline bleaching solution at a temperature ranging from about 80°C to about 90°C; and

(e) washing the papermaking pulp to form a papermaking pulp having a brightness of at least about 65% ISO.

36. The process of claim 22, further comprising treating the papermaking pulp with a bleaching solution at a temperature ranging from about 80°C to about 90°C.

37. The process of claim 36, further comprising washing the papermaking pulp after treating with the bleaching solution.

38. The process of claim 36, further comprising mechanically shearing the papermaking pulp during the treating with the bleaching solution to form a papermaking pulp having a brightness of at least about 65% ISO.

39. (Canceled).

40. The process of claim 38, further comprising mechanically shearing the pulp to a freeness of about 350 ml CSF at the lowest.

41. The process of claim 11, wherein the acid solution further comprises a chelating agent.

42. The process of claim 31, wherein the acid solution further comprises a chelating agent.

43. A process for producing a pulp suitable for making paper from a nonwood fiber source material, the process comprising the steps of:

(a) providing a nonwood fiber source material comprising bast and core fibers;

(b) washing the nonwood fiber source material with a washing solution;

(c) impregnating the nonwood fiber source material with an alkaline pulping solution at substantially ambient temperature and at atmospheric pressure;

(d) feeding the impregnated nonwood fiber source material into a mechanical shearing system at ambient temperature;

(e) initiating mechanical shearing of the nonwood fiber source material in the mechanical shearing system and then neutralizing residual alkali in the impregnated nonwood fiber source material by addition of an acid solution; and

(f) continuing the mechanical shearing to form a papermaking pulp having a freeness of at least about 250 ml CSF and having a brightness of at least about 55% ISO.

44. The process of claim 43, wherein the acid solution further comprises a chelating agent.

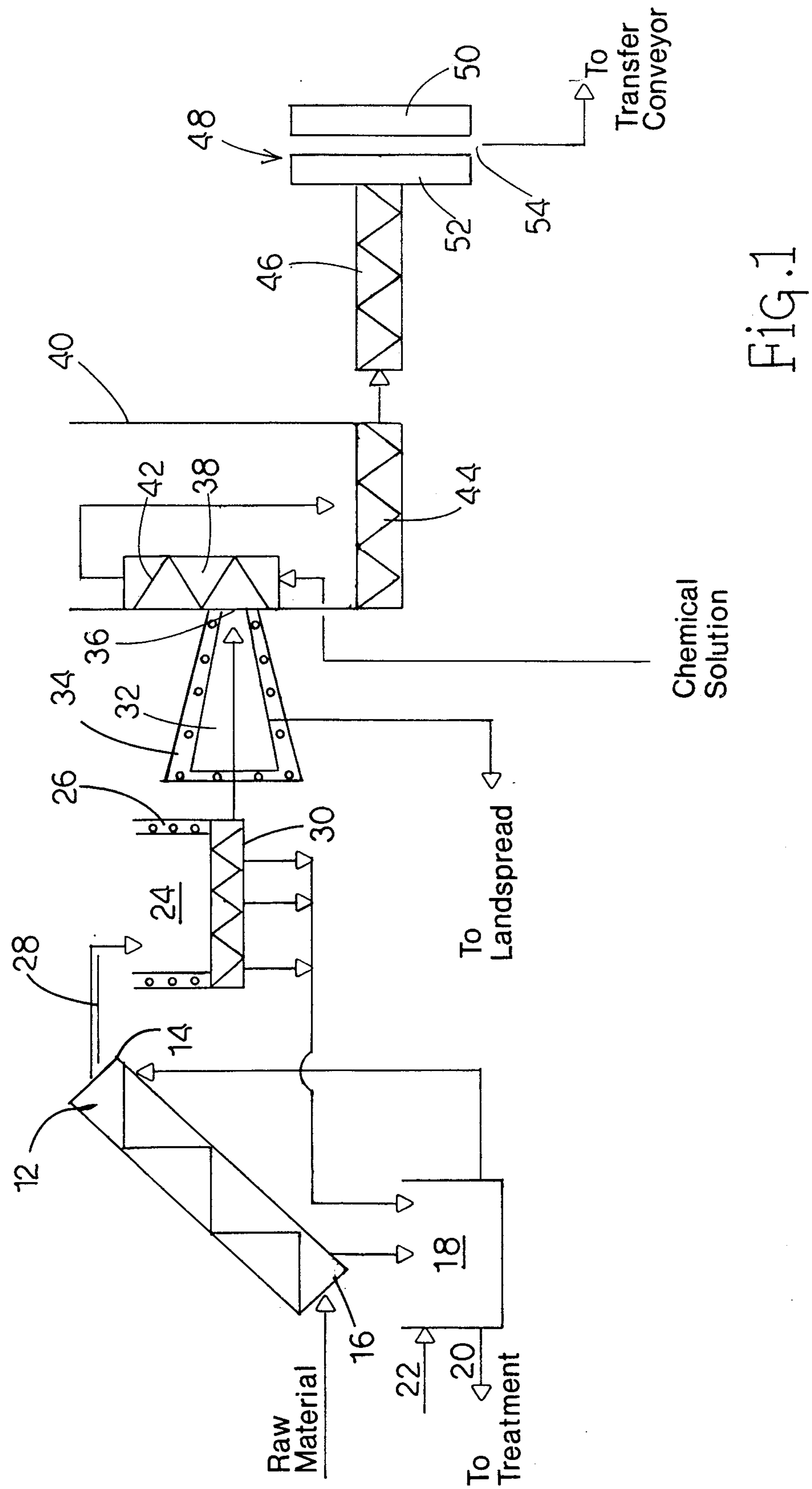

Raw Material
To Treatment
To Landspread
Chemical Solution
To Transfer Conveyor
12
14
16
18
20
22
24
26
28
30
32
34
36
38
40
42
44
46
48
50
52
54

FIG.1

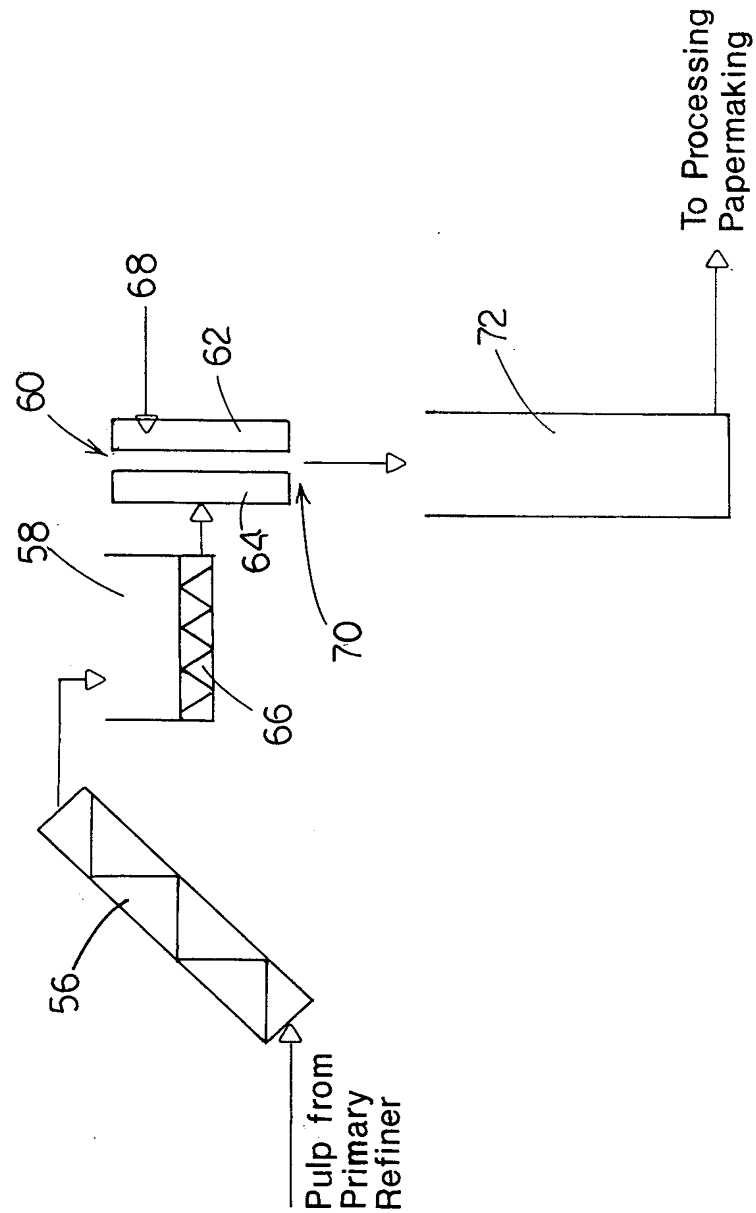

Pulp from Primary Refiner
56
58
60
62
64
66
68
70
72
To Processing Papermaking

FIG. 2

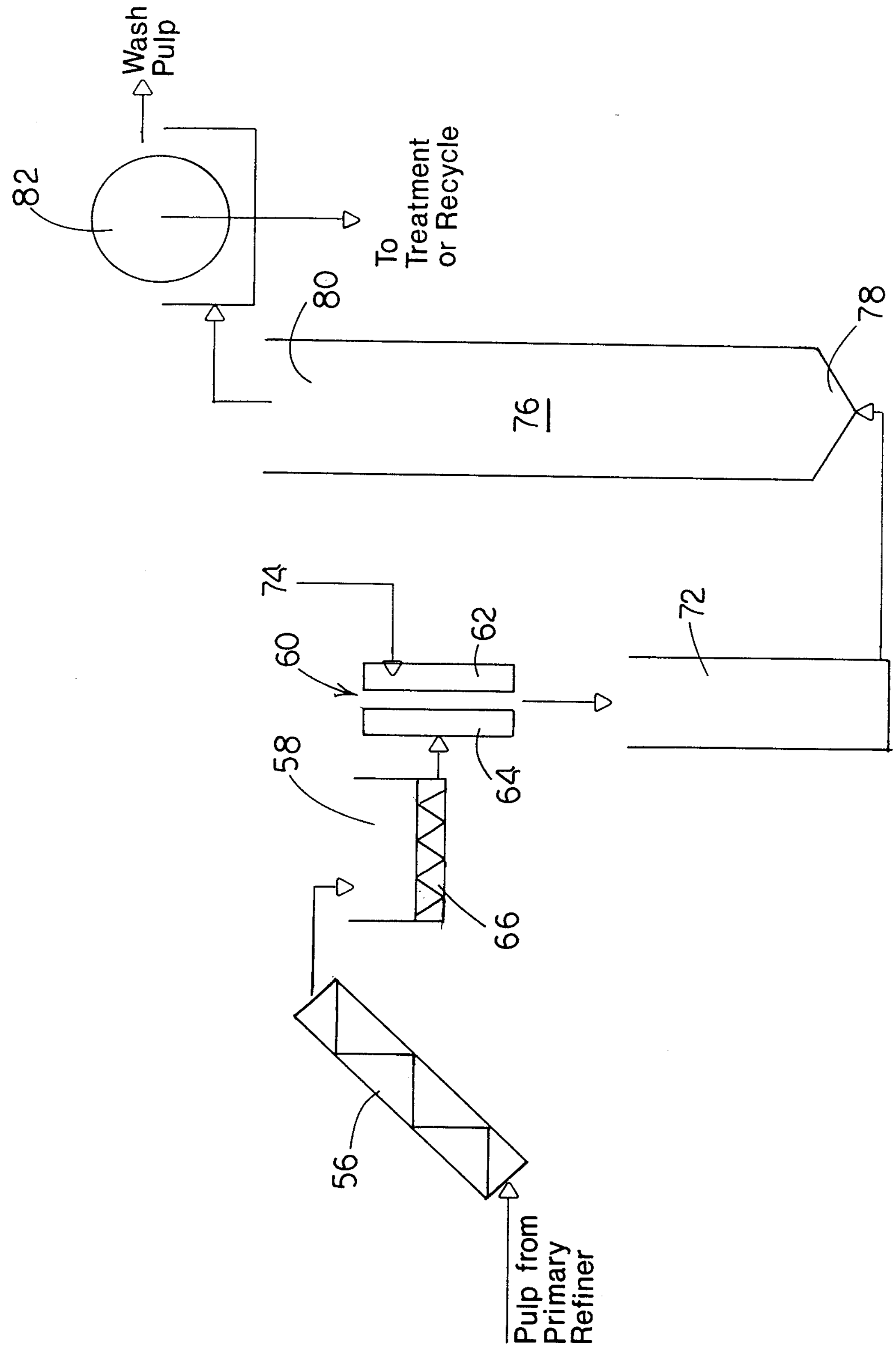

Wash
Pulp
82
To
Treatment
or Recycle
80
76
78
74
60
62
64
72
58
66
56
Pulp from
Primary
Refiner

FIG. 3

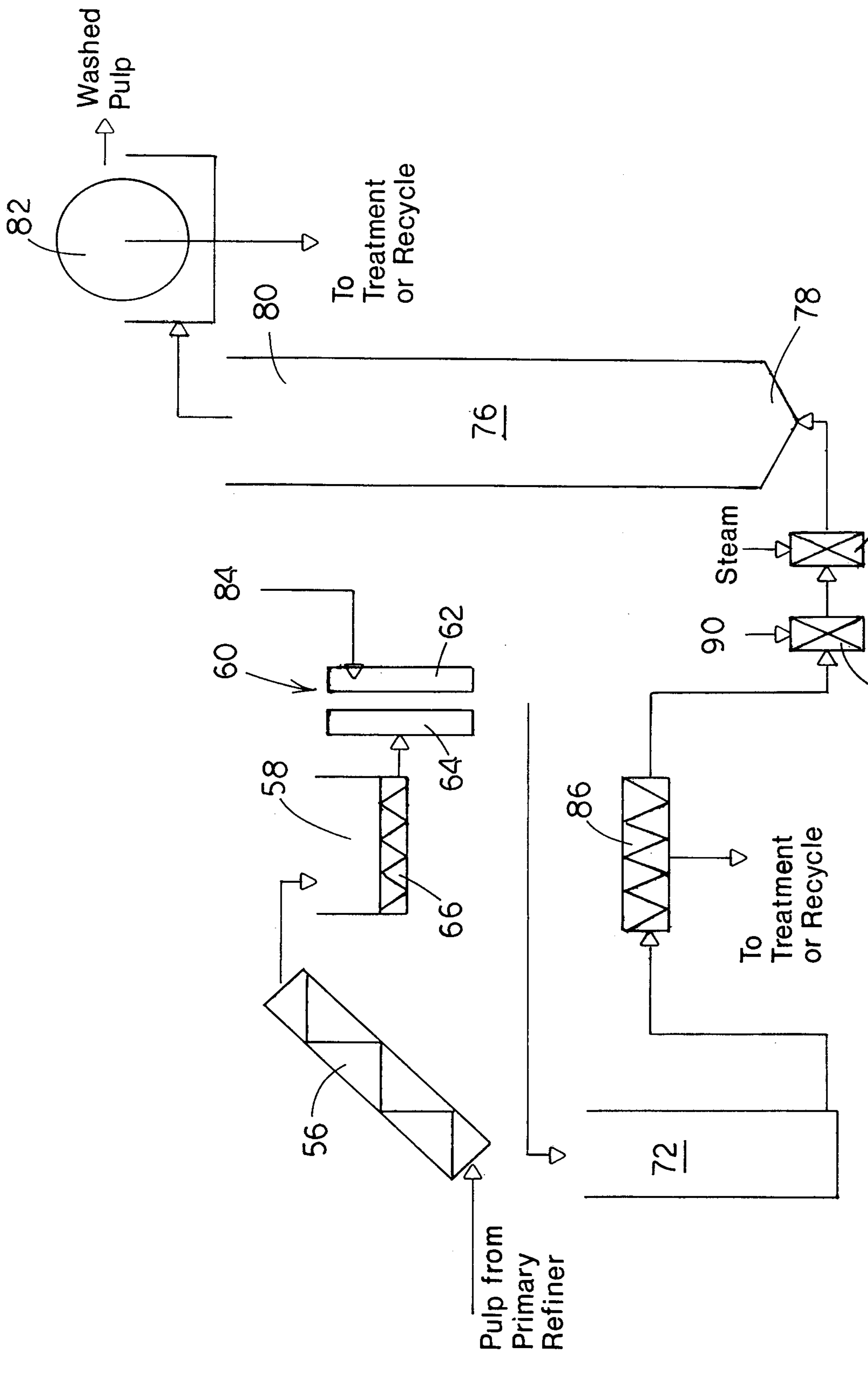

Washed Pulp
82
To Treatment or Recycle
80
76
78
92
Steam
90
88
84
60
62
64
58
66
56
86
To Treatment or Recycle
72
Pulp from Primary Refiner

FIG.4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/02643

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :D21B 1/02, 1/04; D01C 1/00
US CL :162/24, 26, 27, 60, 96, 97, 98
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 162/24, 26, 27, 60, 96, 97, 98

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
APS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ---- Y	US 5,002,635 A (GENTILE, Jr. et al) 26 March 1991, col. 4, line 65; col. 5, line 10; col.6, lines 9-15, 36-44, 59, 60, 64; col. 8, lines 36-46, 57-64.	1,2, 4-10, 12, 15, 22, 23, 25-30, 32 ---- 3, 11, 13, 14, 16-21, 24, 31, 33-40

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

27 MARCH 1999

Date of mailing of the international search report

26 APR 1999

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

STEVEN LEAVITT

Telephone No. (703) 305-6929